

U.S. Patent Application Serial No. 09/938,310

Amendment dated January 8, 2004

Reply to OA of July 11, 2003

**REMARKS**

Claims 1-3, 15-24 and 35 are pending in this application. Claims 1 and 2 have been canceled herein without prejudice or disclaimer. Non-elected claims 5-6, and 25-34, have been canceled.

Claims 3, 15, 16, 18-22, 24, and 35 have been amended herein. No new matter has been added by this amendment.

In view of the amendments to the claims and the remarks set forth below, further and favorable reconsideration is respectfully requested.

*I. Claims 1-3, 15-24 and 35 are rejected under 35 U.S.C. §112, first paragraph, because the specification, while being enabling for purification of vinyl polymers obtained by atom transfer polymerization (ATRP), using the exemplified oxidizing agents, does not reasonably provide enablement for purification using the scope of oxidizing agents set forth in the specification and claims.*

Claims 1 and 2 have been canceled without prejudice.

Claim 18 has been amended to limit the scope of the oxidizing agent, to an oxidizing agent that results in the production of a purified vinyl polymer.

Amended claim 18 positively recites that purified polymer is obtained, and thus excludes any and all inoperative embodiments.

The determination of whether the use of a particular oxidizing agent results in the purified polymer can be readily made by the skilled artisan without undue experimentation, using only routine screening techniques. Routine experimentation does not constitute “undue experimentation.”

The Examiner states that many of the agents set forth as suitable oxidizing agents are known

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to cause reactions of vinyl group containing polymers including polymerization, cross-linking, grafting, etc. Again, it is noted that the claims as amended, encompass only those agents that produce the purified polymer.

In view of the foregoing remarks and claims as amended, it is submitted that the specification enables the full scope of the invention as presently claimed, within the meaning of 35 USC § 112, first paragraph. Accordingly, the Examiner is respectfully requested to withdraw this rejection.

**II. *Claims 1-3, 15-24 and 35 are rejected under 35 U.S.C. §103(a) as being unpatentable over Roos in view of Nakagawa.***

Claims 1 and 2 have been canceled without prejudice. Claim 18 has been amended to require production of a purified polymer. Claims 3, 15-24 and 35 have been amended as appropriate, to be directly or indirectly dependent on amended claim 18.

The Examiner states that the issue is not whether the references should be combined, but that when combined, whether the skilled artisan would have found the combination obvious.

Under United States Patent Law, there must be some motivation, suggestion or incentive, supporting the combination of references, or the combination is improper. This motivation must come from the references themselves.

Thereafter, the properly combined references must provide some motivation to the skilled artisan to modify their disclosure to obtain the claimed invention., and there must be a reasonable expectation of success.

In the present case, it is submitted that the combination of Roos with Nakagawa is improper

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because Roos does not suggest vinyl polymers that can be used in curable compositions, and thus the skilled artisan would not be motivated to look to art concerned with vinyl polymer for use in curable compositions (Nakagawa). Likewise the skilled artisan in view of Nakagawa which does not suggest the need for further purification or the need to remove residual metal, would not be motivated to look to art concerned with the removal of transition metals.

Assuming arguendo, the combination proper, neither Roos nor Nakagawa provide any motivation to modify their disclosed methods/polymers to purify the polymers of Nakagawa using the purification method of Roos. Example 5 of Nakagawa teaches synthesis of a silyl-terminated poly(n-butyl acrylate) using a platinum catalyst where the catalyst is not removed, and the polymer produced has a number average molecular weight of 13,900 and a molecular weight distribution of 1.25. In Reference Example 5, the silyl-terminated polymer produced is molded into a uniform cured sheet having good breaking strength and elongation. Thus, the skilled artisan in view of Nakagawa would have no motivation to employ a purification process to remove the platinum catalyst, because Nakagawa teaches that a polymer suitable for use is obtained without removal of a platinum catalyst and Nakagawa teaches that simple production methods are desirable.

Further, regarding independent claim 18, it is submitted that the present invention is not obvious over *Roos* in view of *Nakagawa*, since *Roos* does not teach or suggest that the vinyl polymer obtained may be used as a component for a hydrosilylatable composition, as presently required.

As is described on page 59, lines 7-11, in the instant specification, a vinyl polymer treated with the present invention has an increased hydrosilylation activity, and can be used as a component

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of a hydrosilylation composition. Neither of Roos nor Nakagawa teach or suggest increased hydrosilylation activity.

Attached hereto please find a Declaration under 37 CFR rule 1.132, illustrating the superior hydrosilylation activity of the present invention. According to the results of the illustrated experiment, the amount of a platinum catalyst necessary for gelation, which is an indicator of hydrosilylation activity, was decreased after the air oxidation treatment of the present invention, in addition to the reduction of remaining copper content. This establishes that the present purification method enhances hydrosilylation activity of a hydrosilylatable composition.

In view of the amendments to the claims, the remarks set forth above and the attached Declaration, it is submitted that nothing in Roos or Nakagawa, taken alone or together, render the claimed invention obvious within the meaning of 35 USC § 103. Accordingly, the Examiner is respectfully requested to withdraw this rejection.

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If, for any reason, it is felt that this application is not now in condition for allowance, the Examiner is requested to contact Applicants undersigned attorney at the telephone number indicated below to arrange for an interview to expedite the disposition of this case.

In the event that this paper is not timely filed, Applicants respectfully petition for an appropriate extension of time. Please charge any fees for such an extension of time and any other fees which may be due with respect to this paper, to Deposit Account No. 01-2340.

Respectfully submitted,

ARMSTRONG, KRATZ, QUINTOS, HANSON & BROOKS, LLP



Susanne M. Hopkins  
Attorney for Applicant  
Reg. No. 33,247

SMH/plb  
Atty. Docket No. **011059**  
Suite 1000  
1725 K Street, N.W.  
Washington, D.C. 20006  
(202) 659-2930



**23850**

PATENT TRADEMARK OFFICE

Enclosures: Declaration under 37 C.F.R. 1.132

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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of:

Fujita et al. *JAN 08 2004*

Serial No.: 09/938,310 *SEARCH & TRADEMARK OFFICE* Art Unit: 1713

Filed : August 24, 2001

Examiner: WILSON, DONALD R

Title : PURIFICATION WITH OXIDIZING AGENTS OF VINYL  
POLYMERS OBTAINED BY ATOM TRANSFER RADICAL POLYMERIZATION

**RECEIVED**

JAN 14 2004

**TC 1700**

DECLARATION UNDER RULE 132

Honorable Commissioner of Patents and Trademarks,  
Washington, D.C. 20231

Sir:

I, Akihisa Hirota, a citizen of Japan and having  
postal mailing address of KANEKA CORPORATION Osaka  
Plant, 5-1-1, Trigainishi, Settsu-shi, Osaka 566-0072  
JAPAN, declare and say that:

March, 1992, I was graduated from Yokohama National  
University, Faculty of Engineering, Division of Materials  
Science;

From April, 1992, up till the present, I have been  
employed by Kaneka Corporation;

From May, 1992 till February, 1995, I was engaged in  
the works of study of polymerization processes of  
polystyrene for improving productivity;

From March, 1995 till April, 1999, I was engaged in  
the works of studies of purification processes of  
polyisobutylene and production methods of an initiator  
used in an isobutylene polymerization;

From April, 1999 till November, 2000, I was engaged  
in the works of study of production methods of

styrene/isobutylene/styrene block copolymer; and

From April, 1988, up till the present, I have been engaged in the works of studies of purification process of polyacrylate;

I am familiar with the subject matter thereof;

I have read the Official Action mailed and the references cited therein and am familiar with the subject matter thereof;

I respectfully submit herewith my exact report thereon;

In order to demonstrate that the method of the present invention makes it possible to increase hydrosilylation activity, I have carried out the following experiment.

<Production example 1>

In the followings, liquid materials to be used were deoxidized in advance.

A 250-L pressure reaction vessel was charged with 1.76 kg (12.3 mol) of copper(I) bromide followed by nitrogen purging. Then, acetonitrile (18.5 kg) was added and the mixture was stirred at about 70°C for 35 minutes. Then, 3.69 g (10.2 mol) of diethyl 2,5-dibromo adipate and 42.0 kg (328 mol) of butyl acrylate were added thereto. Then, 71.0 g (0.410 mol) of pentamethyldiethylenetriamine (hereinafter briefly as triamine) was added to thereby start the reaction, and the reaction mixture was heated and stirred the mixture with controlling the temperature of the reaction mixture to be between 80 and 90°C. Triamine, 101.5 g (0.585 mol) was further added into the mixture. Triamine, 71.0 g (0.410 mol) was further added twice, and at 45 minutes after the start of reaction, 168

kg (1,310 mol) of butyl acrylate was added dropwise intermittently over 165 minutes. During this operation, 71.0 g of triamine (0.410 mol) was further added three times. At 275 minutes after the start of reaction, 31.6 kg of the reaction mixture was taken out from the reaction vessel, and then 16.5 kg (150 mol) of 1,7-octadiene and 608 g (3.51 mol) of triamine were added, and the mixture was kept heating and stirring at 80°C. At 330 minutes after the addition of octadiene, 608 g (3.51 mol) of triamine was further added. At 195 minutes after the addition of triamine, heating was stopped. Volatile matters were removed under reduced pressure and heated condition, to give polymer compound [1]. By GPC analysis (mobile phase: chloroform, polystyrene equivalence method), the polymer [1] was found to have a number average molecular weight of 26,700 and a molecular weight distribution of 1.36. The average number of alkenyl groups introduced per molecule of the polymer as determined by <sup>1</sup>H NMR analysis was 1.9.

**<Experimental Examples 1>**

Under air atmosphere, 100 parts (parts by weight; the same applies hereinafter) of toluene is added to 100 parts of the polymer [1] and the solid fraction was removed by centrifugation. Kyowaad 500SH (2 parts, product of Kyowa Chemical) and Kyowaad 700 SL (2 parts, product of Kyowa Chemical) was added to the obtained solution, and the mixture was stirred at 100°C for 2 hours. The solution was cooled and then solid fraction was removed by centrifugation under air atmosphere. Volatile matters were removed at 100°C under reduced pressure, to give polymer compound [2].

**<Comparative Experimental Examples 1>**

Under nitrogen atmosphere, 100 parts (parts by weight; the same applies hereinafter) of toluene is added to 100 parts of the polymer [1] and the solid fraction was removed by centrifugation with keeping nitrogen atmosphere. Kyowaad 500SH (2 parts, product of Kyowa Chemical) and Kyowaad 700 SL (2 parts, product of Kyowa Chemical) was added to the obtained solution, and the mixture was stirred under nitrogen atmosphere at 100°C for 2 hours. The solution was cooled and then solid fraction was removed by centrifugation with keeping nitrogen atmosphere. Volatile matters were removed at 100°C under reduced pressure, to give polymer [2'].

(Quantitative determination of copper)

The polymer was mixed with super-high purity nitric acid and super-high purity sulfuric acid and subjected to microwave decomposition. Using an ICP mass spectrometer (manufactured by Yokogawa Analytical Systems; HP-4500), the residual copper in the decomposition product was assayed to determine the residual copper in the polymer.

Furthermore, gelation times at 130°C were determined on the polymers obtained in Experimental Examples 1 and Comparative Experimental Examples 1 (Curing test). The results are shown in Table 1.

(Curing test)

The polymer was manually blended with a linear polymethylsiloxane containing an average of five hydrosilyl groups and an average of five  $\alpha$ -methylstyrene residues (specifically, 2-phenylpropyl group, 1-methyl-1-phenylehtyl group) (Si-H value: 3.70 mol/g) and a solution of platinum(0)-1,1,3,3-tetramethyl-1,3-divinyldisiloxane complex in xylene (platinum concentration  $1.3 \times 10^{-5}$  mmol/ $\mu$ l) at room temperature to give a composition. About 0.1 g of this composition was

heated with stirring in atmospheric air on a hot plate at 130°C and the time to gelation (gelation time, in seconds) was measured.

The linear polymethylsiloxane mentioned above was used in such an amount as to give an alkenyl/hydrosilyl molar ratio of 1/1.5. The amounts of the platinum catalyst as shown in Table 1 are the equivalent amounts (molar ratios) of the platinum catalyst relative to the alkenyl group.

Table 1

Conditions	Cu content (ppm)	Gelation time (sec.)	
		(The amount of platinum catalyst = $5 \times 10^{-3}$ equiv.)	(The amount of platinum catalyst = $2 \times 10^{-2}$ equiv.)
Under air atmosphere	75	30	No Data
Under nitrogen atmosphere	358	The polymer could not be cured	The polymer could not be cured

RESULTS

As is shown above, copper content of the polymer was reduced by carrying out an air oxidation treatment of the polymer. Moreover, the amount of a platinum catalyst necessary for gelation, which is an indicator of hydrosilylation activity, was decreased after the air oxidation treatment.

I declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Signed this 25<sup>th</sup> day of December , 2003

Akihisa Hirota

Akihisa Hirota